

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**

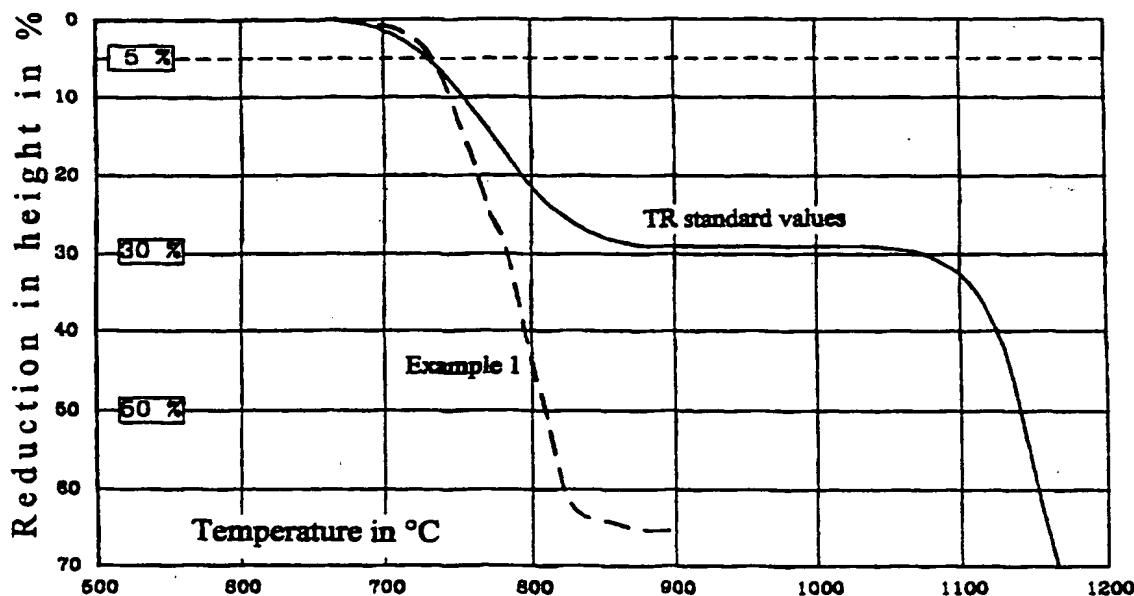


INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C03C 13/00, 13/06		A1	(11) International Publication Number: WO 96/04213
			(43) International Publication Date: 15 February 1996 (15.02.96)
(21) International Application Number: PCT/EP95/02374		(81) Designated States: AU, BR, CA, CN, CZ, FL, HU, IS, JP, KR, NO, NZ, PL, SI, SK, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).	
(22) International Filing Date: 19 June 1995 (19.06.95)			
(30) Priority Data: P 44 27 368.1 2 August 1994 (02.08.94) DE 195 03 168.7 1 February 1995 (01.02.95) DE		Published With international search report.	
(71) Applicant (for all designated States except US): ISOVER SAINT-GOBAIN [FR/FR]; Les Mirois, 18, avenue d'Alsace, F-92400 Courbevoie (FR).			
(72) Inventors; and (75) Inventors/Applicants (for US only): LOHE, Peter [DE/DE]; Ritterstrasse 5, D-67112 Mutterstadt (DE). HOLSTEIN, Wolfgang [DE/DE]; Herderstrasse 2, D-35315 Homberg (DE). SCHWAB, Wolfgang [DE/DE]; Schönaauer Strasse 25, D-68723 Plankstadt (DE).			
(74) Agent: KADOR & PARTNER; Corneliusstrasse 15, D-80469 Munich (DE).			

(54) Title: A MINERAL-FIBER COMPOSITION

Temperature behaviour (Swedish method)



(57) Abstract

A biologically degradable mineral-fiber composition characterized by the following constituents in percent by weight: SiO₂: 40 to less than 52; Al₂O₃: less than 4; CaO: more than 25 and up to 45; MgO: 5 to 15; BaO: 0 to 7; Na₂O: 2 to 12; K₂O: 0 to 10; Na₂O + K₂O: 2 to 15; TiO₂, Fe₂O₃, MnO: 0 to 5.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	GB	United Kingdom	MR	Mauritania
AU	Australia	GE	Georgia	MW	Malawi
BB	Barbados	GN	Guinea	NE	Niger
BE	Belgium	GR	Greece	NL	Netherlands
BF	Burkina Faso	HU	Hungary	NO	Norway
BG	Bulgaria	IE	Ireland	NZ	New Zealand
BJ	Benin	IT	Italy	PL	Poland
BR	Brazil	JP	Japan	PT	Portugal
BY	Belarus	KE	Kenya	RO	Romania
CA	Canada	KG	Kyrgyzstan	RU	Russian Federation
CF	Central African Republic	KP	Democratic People's Republic of Korea	SD	Sudan
CG	Congo	KR	Republic of Korea	SE	Sweden
CH	Switzerland	KZ	Kazakhstan	SI	Slovenia
CI	Côte d'Ivoire	LI	Liechtenstein	SK	Slovakia
CM	Cameroon	LK	Sri Lanka	SN	Senegal
CN	China	LU	Luxembourg	TD	Chad
CS	Czechoslovakia	LV	Latvia	TG	Togo
CZ	Czech Republic	MC	Monaco	TJ	Tajikistan
DE	Germany	MD	Republic of Moldova	TT	Trinidad and Tobago
DK	Denmark	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	US	United States of America
FI	Finland	MN	Mongolia	UZ	Uzbekistan
FR	France			VN	Viet Nam
GA	Gabon				

A mineral-fiber composition

The present invention relates to a mineral-fiber composition that is biologically degradable.

The prior art describes some mineral-fiber compositions which are said to be biologically degradable.

The biological degradability of mineral-fiber compositions is of great importance because various studies point out that mineral fibers with very small diameters in the range of less than 3 microns are suspected to be carcinogenic, while biologically degradable mineral fibers of such dimensions show no carcinogenicity.

However the mineral fiber compositions must also be easy to process by known methods for making mineral wool with a small diameter, in particular the jet process. This means in particular a sufficient processing range of for example 80°C and suitable viscosity of the glass melt.

Also, the mechanical and thermal properties of the mineral fibers, or the products produced therefrom, are of crucial importance. For example mineral fibers are used to a great extent for insulation purposes. Particularly for use in the industrial sector, sufficient temperature resistance of the mineral fibers is necessary.

The invention is based on the problem of providing a novel mineral-fiber composition that is characterized by biological degradability, has good temperature resistance and is easy to process.

The invention is based on the finding that this problem can be solved by a mineral-fiber composition that consists substantially of silicon dioxide and alkaline-

earth oxides, and also contains sodium oxide and/or potassium oxide as a melting accelerator and a sizable amount of aluminum oxide to increase the temperature resistance.

It has turned out that such mineral-fiber compositions fulfill the combination of the necessary properties, namely biological degradability, sufficient temperature resistance for insulated objects in industry, and good processibility for producing the mineral wool as such and the products. This means at the same time that the upper devitrification temperature of the melt is preferably under 1300°C. The mean fiber diameter is preferably 10 microns or less, and is in particular between 2.5 and 5 microns.

The object of the invention is a mineral-fiber composition that is biologically degradable, characterized by the following constituents in percent by weight:

SiO ₂	40 to less than 52
Al ₂ O ₃	less than 4
CaO	more than 25 and up to 45
MgO	5 to 15
BaO	0 to 7
Na ₂ O	2 to 12
K ₂ O	0 to 10
Na ₂ O + K ₂ O	2 to 15
TiO ₂ , Fe ₂ O ₃ , MnO	0 to 5.

The inventive mineral-fiber compositions are in particular easy to draw by the jet process, i.e. one obtains a fine, low-slug mineral wool.

The mineral fibers reach a high temperature resistance of at least 730°C.

The mineral fibers show high biological degradability.

The addition of sodium oxide and/or potassium oxide lowers the melting point, thereby improving processability in the melting process. Also, if the mineral-wool composition contains sodium it is advantageous to use up to 35% broken waste glass.

The inventive mineral-fiber compositions can preferably be melted at melting temperatures of 1350 to 1450°C in melting chambers fired by fossil fuels, in particular natural gas. Such melting chambers can produce a homogeneous melt, which is the precondition for constant product quality. Homogeneity of the glass melt also facilitates reproducibility of the fiber-forming process and thus the thermal and mechanical product properties. Furthermore, the constant chemical composition of the thus produced mineral wool leads to controllable biological degradability.

In particular the addition of aluminum oxide increases the temperature resistance of the mineral wool.

The inventive mineral-fiber compositions preferably have the following constituents in percent by weight:

SiO ₂	40	to 51.5
Al ₂ O ₃	2 to less than 4	
CaO	25.5	to 40
MgO	8	to 15
BaO	0	to 5
Na ₂ O	3	to 8
K ₂ O	2	to 10
Na ₂ O + K ₂ O	5	to 10
TiO ₂ , Fe ₂ O ₃ , MnO	0	to 3.

A content of silicon oxide in the range of 40 to 55 percent by weight is particularly preferred.

With respect to the alkali oxides a range of 5 to 8 percent by weight is particularly preferred. Aluminum oxide is preferably present in a range between 3 and 4 percent by weight.

Barium oxide, which can be used instead of calcium or magnesium oxide, is present in an amount up to 7 percent by weight, preferably up to 5 percent by weight, in particular 0.5 to 3 percent by weight.

Sodium oxide is preferably present in an amount of more than 2 percent by weight.

An aluminum oxide content between 1 and 2 percent by weight, in particular of 1.5 percent by weight, is also particularly preferred.

The content of iron is preferably 0.5 to 2.5 percent by weight.

To assess biological degradability the standard powder test of the German Glass Society was used. This is an easily conducted method and gives a sufficient measure of biological degradability when used with a simulated physiological lung fluid at 37°C. The method is described in L. Springer, "Laboratoriumsbuch für die Glasindustrie", 3rd edition, 1950, Halle/S: W. Knapp Verlag.

The temperature behavior of the mineral fibers was determined by the "Swedish method". In this method a silit tube furnace is used with a horizontal working tube open on both sides having a length of 350 mm and an inside diameter of 27 mm. In the center of the furnace there is a small

ceramic supporting plate (30 X 20 X 3 mm) for holding the test sample. The test sample has dimensions of 12 X 12 X 12 mm or 12 mm \varnothing X 12 mm height. The bulk density is normally 100 kg/m³. The temperature increase is 5 K/min. The change in test sample height is determined continuously with a reading optic.

The invention shall be described in more detail in the following with reference to examples.

Example 1

A mineral wool was produced with the following composition in percent by weight:

SiO ₂	51
Al ₂ O ₃	3
Fe ₂ O ₃	0.3
CaO	31
MgO	10
Na ₂ O	5
K ₂ O	0.1.

This composition could be processed well to mineral fibers with a mean diameter of 2.0 to 10 microns by the jet process at a drawing temperature between 1300 and 1400°C.

An investigation according to the standard powder test of the German Glass Society yielded a value of 40 mg/kg and thus a value for high biological degradability.

Determination of temperature behavior by the Swedish method yielded a temperature resistance at 5% reduction in height of 735°C, which can be clearly seen in the

corresponding diagram shown by way of example in the single drawing.

Example 2

A mineral wool was produced with the following composition in percent by weight:

SiO ₂	46.5
Al ₂ O ₃	3.5
CaO	35
MgO	10
Na ₂ O	5.

This composition could also be processed well to mineral fibers with a mean diameter of 2.0 to 10 microns by the jet process at a drawing temperature between 1300 and 1400°C.

An investigation according to the standard powder test of the German Glass Society yielded a value of 35 mg/kg and thus a value for high biological degradability.

Determination of temperature behavior by the Swedish method yielded a temperature resistance at 5% reduction in height of 800°C.

This example shows that glass with a high aluminum oxide content has excellent temperature resistance, which is in turn a criterion for the fire resistance of the products.

Example 3

A mineral wool was produced with the following composition in percent by weight:

SiO ₂	50
Al ₂ O ₃	2.8
Fe ₂ O ₃	0.9
CaO	25.6
MgO	9.7
Na ₂ O	4.9
K ₂ O	1.0
BaO	4.9.

This composition could be processed well to mineral fibers with a mean diameter of 2.5 to 10 microns by the jet process at a drawing temperature between 1300 and 1400°C.

Example 4

A mineral wool was produced with the following composition in percent by weight:

SiO ₂	51
Al ₂ O ₃	2.6
Fe ₂ O ₃	1.1
CaO	27.9
MgO	10.4
Na ₂ O	3.2
K ₂ O	0.6
BaO	3.1.

This composition could be processed well to mineral fibers with a mean diameter of 2.5 to 10 microns by the jet process at a drawing temperature between 1300 and 1400°C.

Example 5

A mineral wool was produced with the following composition in percent by weight:

SiO ₂	50.9
Al ₂ O ₃	2.6
Fe ₂ O ₃	1.1
CaO	30
MgO	10.4
Na ₂ O	2.2
K ₂ O	1.6
BaO	1.1.

This composition could be processed well to mineral fibers with a mean diameter of 2.5 to 10 microns by the jet process at a drawing temperature between 1300 and 1400°C.

Claims

1. A mineral-fiber composition that is biologically degradable, characterized by the following constituents in percent by weight:

SiO ₂	40 to less than 52
Al ₂ O ₃	less than 4
CaO	more than 25 and up to 45
MgO	5 to 15
BaO	0 to 7
Na ₂ O	2 to 12
K ₂ O	0 to 10
Na ₂ O + K ₂ O	2 to 15
TiO ₂ , Fe ₂ O ₃ , MnO	0 to 5.

2. The mineral-fiber composition of claim 1, characterized by the following constituents in percent by weight:

SiO ₂	40 to 51.5
Al ₂ O ₃	2 to less than 4
CaO	25.5 to 40
MgO	8 to 15
BaO	0 to 5
Na ₂ O	3 to 8
K ₂ O	2 to 10
Na ₂ O + K ₂ O	5 to 10
TiO ₂ , Fe ₂ O ₃ , MnO	0 to 3.

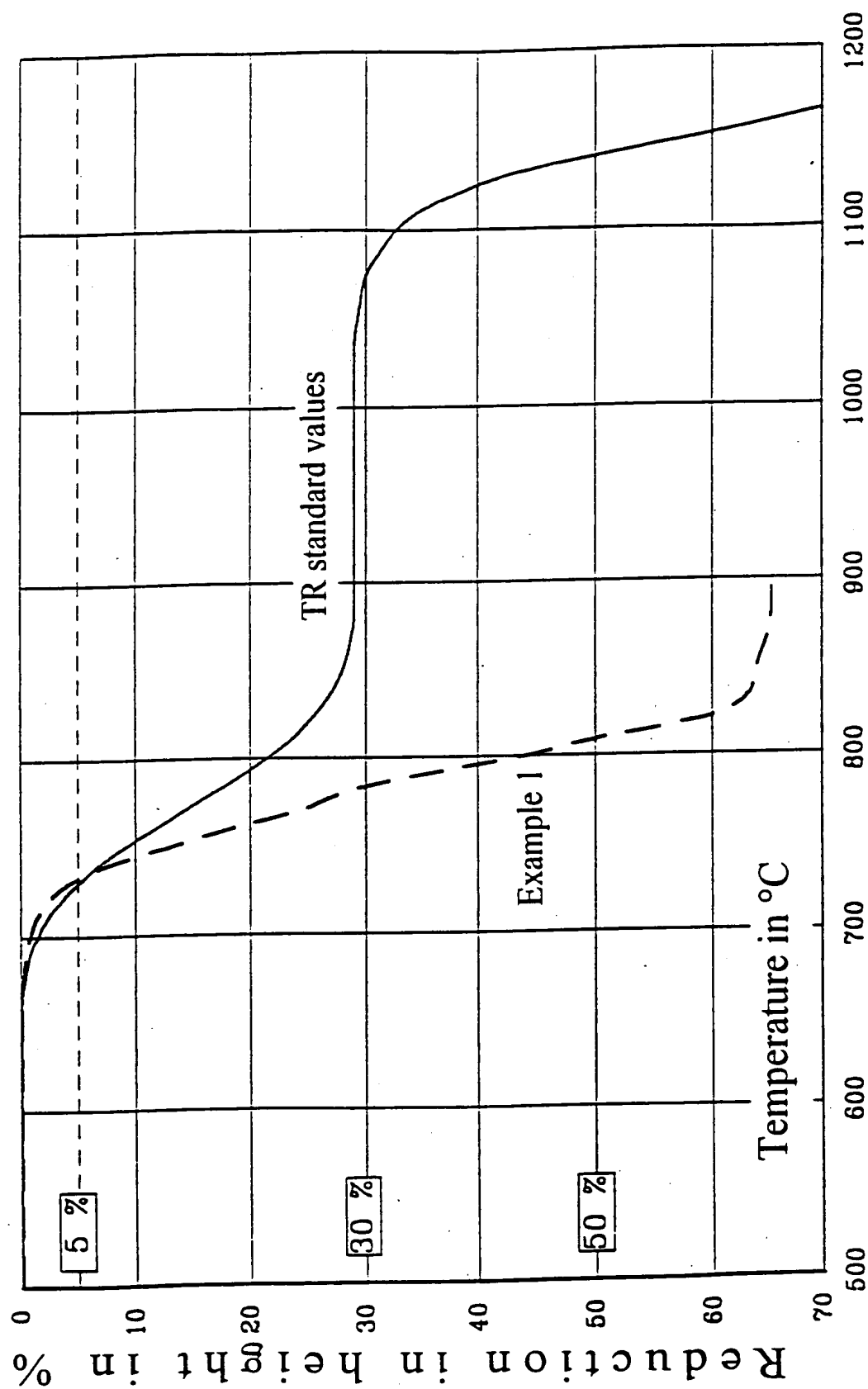
3. The mineral-fiber composition of claim 1 or 2, characterized in that the alkali oxides are present in an amount of 5 to 8 percent by weight.

4. The mineral-fiber composition of any of claims 1 to 3, characterized in that aluminum oxide is present in a content between 3 and 4 percent by weight.

5. The mineral-fiber composition of any of claims 1 to 4, characterized in that the content of iron is 0.5 to 2.5 percent by weight.

6. The mineral-fiber composition of any of claims 1 to 5, characterized in that the content of barium oxide is 0.5 to 4 percent by weight.

Temperature behaviour (Swedish method)



INTERNATIONAL SEARCH REPORT

Internal Application No
PCT/EP 95/02374A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C03C13/00 C03C13/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C03C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	FR,A,2 690 438 (ISOVER SAINT-GOBAIN) 29 October 1993 see page 1, line 37 - page 3, line 21 ---	1-6
X	WO,A,92 09536 (PAROC OY AB) 11 June 1992 see page 1, line 35 - page 4, line 3 ---	1-6
X	EP,A,0 459 897 (ISOVER SAINT-GOBAIN) 4 December 1991 see column 2, line 11 - column 3, line 15 -----	1-6

☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

3 October 1995

Date of mailing of the international search report

20. 10. 95

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl,
Fax (+ 31-70) 340-3016

Authorized officer

Van Bommel, L

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 95/02374

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
FR-A-2690438	29-10-93	AU-B- 4263293	29-11-93
		BR-A- 9305492	11-10-94
		CA-A- 2110998	11-11-93
		CN-A- 1078708	24-11-93
		CZ-A- 9302865	19-10-94
		EP-A- 0596088	11-05-94
		WO-A- 9322251	11-11-93
		HU-A- 67212	28-03-95
		JP-T- 6508600	29-09-94
		NO-A- 934725	20-12-93
		SI-A- 9300218	31-12-93
WO-A-9209536	11-06-92	FI-B- 93346	15-12-94
		AT-T- 117662	15-02-95
		AU-A- 8908791	25-06-92
		DE-D- 69107091	09-03-95
		DE-T- 69107091	17-08-95
		EP-A- 0558548	08-09-93
EP-A-459897	04-12-91	FR-A- 2662688	06-12-91
		AT-T- 121378	15-05-95
		AU-B- 642493	21-10-93
		AU-B- 7731891	05-12-91
		CA-A- 2043699	02-12-91
		CN-A- 1059135	04-03-92
		DE-D- 69108981	24-05-95
		JP-A- 4228455	18-08-92
		US-A- 5250488	05-10-93